Workshop on Frontiers in Synchrotron X-Ray Microbeam Diffraction

January 10, 2003

The Workshop on Frontiers in Synchrotron X-Ray Microbeam Diffraction was held on January 10, 2003 in the Hamilton seminar room in the Chemistry Department at BNL, with a large gathering of over 40 people. The purpose of this workshop was to inform the scientific, university, and industrial community of the plans to design and install a state-of-the-art microdiffraction instrument at NSLS mini-gap undulator beamline X13B. This proposal was submitted to the DOE Office of Science towards the end of January and would be operated as a general user facility with an emphasis on nanoscale research. Opportunities in the cutting-edge science that could be accomplished with this instrument were explored, and user input was solicited.

The workshop was introduced and chaired by Dr. Elaine DiMasi (BNL Physics Dept.), who also outlined the motivation for the proposed instrument. The value of cutting-edge user facilities was described from the perspective of BNL management by Dr. Doon Gibbs, Associate Laboratory Director for Basics Energy Sciences (BES). He emphasized the importance to DOE and BNL of interacting with users at an early stage so that they can influence the evolutionary process by which new facilities are developed and built. He described the organizational structure of BNL BES as well as a number of exciting planned projects. Two such projects that will have a great impact on microbeam diffraction science at BNL are the proposed upgrade to the NSLS and the Center for Functional Nanomaterials (CFN). Dr. Gibbs concluded his presentation with a challenge to the audience to come up with ideas about scientific impacts that can be made by the future NSLS.

The CFN was further elaborated upon by NSLS scientist Dr. Ron Pindak. The CFN is one of five new DOE Nanoscale Science Research Centers (NSRCs). The user programs of the five centers were launched at a workshop in Washington DC on February 26-28, 2003. The CFN has six scientific themes that involve interdisciplinary research on diverse systems, which are listed and described on the CFN website, http://www.cfn.bnl.gov/. The instrumentation and capabilities of the CFN are organized into “lab clusters,” such as materials synthesis, proximal probes, nanopatterning, etc. The CFN will be a user facility similar to the NSLS and both user programs will be coordinated through a common user office. Thus, one proposal can allow access to NSLS beamlines as well as CFN lab tools. Two of the NSLS beamlines, a SAXS beamline on X21 and the X13B microdiffraction beamline, will be optimized to service the needs of CFN General Users. The center, in addition to having the infrastructure to support state-of-the-art fabrication facilities, will provide offices and interaction areas for students and postdocs as well as regular staff.

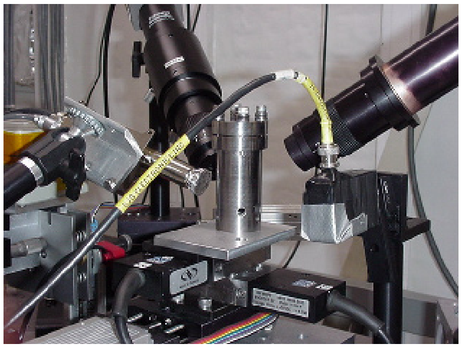
Frontiers in Synchrotron X-Ray Microbeam Diffraction workshop attendees.

Dr. Patricia Mooney of IBM presented a talk on materials research for silicon CMOS technology using microbeam x-ray sources. Strain-relaxed SiGe “virtual substrates” for strained silicon CMOS transistors were described and results of studies characterizing the defect microstructure of these films were presented. Dr. Mooney remarked on how the divergent beam available at X20A of the NSLS limited the resolution at which “micrograins” could be observed, and that a sub-micron, more parallel beam would be of great advantage in her work.

A survey of synchrotron microdiffraction capabilities around the world was given by Prof. Cev Noyan of IBM. He pointed out that several synchrotrons, in particular the ESRF and ALS, have put great emphasis and resources into microbeam diffraction. The great variety in hard x-ray microfocusing optics and a number of designs for accessing reciprocal space (pink beam Laue, scanning monochromator Laue, small sphere-of-confusion six-circle, single-axis, etc.) were explained. The following two talks described in detail the microdiffraction capabilities currently available at the NSLS. Dr. Ken Evans-Lutterodt of Agere described the X16C microdiffraction beamline. He used the example of the selective growth of semiconductor materials on a patterned substrate to illustrate that microdiffraction combined with spectroscopy can be used to obtain strain and chemical composition information with micron-scale spatial resolution. Dr. Jean Jordan-Sweet of IBM next described the capabilities of beamline X20A. This capillary- and diffractometer-based instrument is primarily used to measure strain fields and mosaic structure by scanning diffraction topography. Results were presented on interfacial stress/strain in metal features on silicon and on electromigration-induced stress in narrow metal lines.

The proposed microdiffraction instrument to be built at X13B was described in the next two talks. Dr. James Ablett of the NSLS presented specifications and recently measured spectral plots of the new X13 Mini-Gap Undulator (MGU) source. He then described the plan to use a 4-bounce silicon monochromator and a variety of x-ray microfocusing optics for the proposed instrument. The monochromator can be removed for pink-beam studies, and the optics will be interchangeable between KB mirrors, capillary, pinholes, zone plates, and planar refractive lenses. The modular design will allow for great flexibility in beamsize and divergence selection. Dr. Ken Evans-Lutterodt further described the diffractometer and detector configurations. In order to minimize vibration and torque on the sample and optics, the detector arm will be a completely separate system. The entire microdiffraction instrument is being designed to be robust, easy to align and use, and modular, in order to serve a variety of users from students to busy experts.

After a break for lunch, the workshop resumed with presentations on scientific opportunities by researchers from a range of disciplines. Dr. Mehmet Sarikaya of the University of Washington began with an overview of the fascinating world of structural biomimetics. Biomaterials such as spiders' silk, mother-of-pearl, protein coats on certain bacteria, sea urchin spines, and sponge spicules exhibit nanoorganization. Understanding the structural, functional, and process design characteristics of these self-assembled structures will lead to the invention of engineered "bio-inspired" materials of the future. Next, Dr. DiMasi presented a talk prepared by Dr. Joanna Aizenberg of Lucent Technologies, which described how self-assembled nano structures of calcite crystals and other materials can be formed using organic alkane chain templates which have a variety of attached functional groups. These assemblies of crystals can be patterned and oriented in many ways by changing the functional group, chain tilt, and lithographic pattern. Following this talk, Prof. Valery Kiryukhin of Rutgers University described several correlated electronic systems that form functional materials, which exhibit large changes in electrical or magnetic properties (such as colossal magnetoresistance) under relatively weak external perturbations. He showed an example of strain mapping in the vicinity of a grain boundary taken at beamline 2ID-D at the APS. In order to study these systems, a microdiffraction instrument with temperature control, precise sample positioning, optical access, and tunable energy is needed. The scientific opportunity session was concluded with a talk by Dr. Jeffrey Kysar of Columbia University who discussed the difficulty in determining the relationship between stress and strain in materials. He has simplified the problem by reducing it from 3-D to 2-D by performing experiments using a line of applied force rather than the conventional point indenter on a metal



Microdiffraction setup at beamline X13B.

surface. A microbeam diffraction beamline would allow for the measurement of residual stresses and dislocation density on the surface of laser shock processed samples as a function of position from the "peened" line.

During the afternoon discussion session Dr. DiMasi read letters from members of the Clay Minerals Society email listserver. These researchers would like to see an instrument having high brilliance, a sensitive area detector, variable spot size, and control over temperature and humidity. Audience members also supported the desire for environmental control, citing the need to study self-assembly experiments in liquid solution rather than in just the dried post-assembly state. There is a need to get all information - tilt, chemical, lattice spacing, transformation temperature, etc., from single grains on the order of a micron in size. Many times the need for a parallel beam was mentioned. A discussion about the measurement of organic and bio-materials brought up legitimate concern over the possibility of beam damage. Another desired capability mentioned several times is computed tomography. Questions were asked about the time structure of the existing NSLS x-ray ring vs. the proposed upgrade. Pump-probe experiments are possible now with a ~0.5 nanosecond period, and in the future with ~20 femtosecond resolution. The APS currently has a 50 picosecond timing structure. It was pointed out that single-shot microdiffraction would not yield enough intensity, but a locked-in, repeated pump-probe setup would be able to accumulate enough signal.

The workshop concluded with committee members encouraging researchers to try microbeam experiments now on existing instruments, in order to see what is needed and what works. It is expected that the new microdiffraction beamline will constantly evolve over time, and experience now will lead to a better instrument in the future. Many components are available now or will be soon for experimental trials.

—Jean Jordan-Sweet

Two NSLS Scientists Win Engineering Awards

January 30, 2003

At the BNL Employee Recognition Award Ceremony on January 30, 17 Lab employees were rewarded with BNL's highest honors, including two NSLS scientists who won the $5,000 Engineering Awards, which are given to recognize distinguished contributions to engineering or computing over one or more years.

Donald Lynch, a project engineer who joined BNL in September 1991, and George Rakowsky, an electrical engineer who returned to BNL in July 1993, both at the NSLS, were recognized for their sustained contributions in the development of small-gap, in-vacuum undulators, a technology now adopted at most synchrotron radiation facilities in the world.

Pictured at right with Associate Laboratory Director for Facilities & Operations Mike Bebon (back, right) are the recipients of BNL's 2002 Engineering Award: (back, from left) Donald Lynch, NSLS Department; George Rakowsky, NSLS; Jack Fried, Instrumentation Division; (front, from left) George Ganetis, Magnet Division; Christopher Channing, Plant Engineering Division; and Joseph Levesque, Emergency Services Division.

The NSLS operates two electron storage rings: the 2.8-giga electron volt (GeV) x-ray ring and a 0.8-GeV vacuum ultraviolet ring, which contain periodic magnetic structures called wigglers and undulators, collectively known as insertion devices. Electrons are sent racing around the rings at nearly the speed of light. When they cross an insertion device, they emit a very intense, narrow beam of synchrotron radiation.

Earlier generations of undulators in medium-energy machines such as the NSLS x-ray ring are able to generate only "soft" x-rays, up to about 1 kilo electron volt (keV) of photon energy. The small-gap undulators generate tunable "hard" x-rays in the range of 3 to 20 keV, which are essential for decoding the structure of complex biological molecules in the rapidly expanding field of structural biology. Previously, tunable hard x-ray beams were available only at the very high-energy storage rings, such as the 7-GeV Advanced Photon Source at Argonne National Laboratory.

The idea of introducing a small-gap, short-period undulator in the NSLS x-ray ring was originated in the late 1980s by Sam Krinsky, then NSLS Deputy Chair. Peter Stefan, then NSLS physicist, did the first proof-of-principle tests and developed the conceptual design of the small-gap undulator. Lynch worked with Stefan on the mechanical, structural, thermal, and vacuum design of the first two generations and on the latest version of the device.

Rakowsky proposed the magnetic design in 1991 and built a demonstration model, followed by the full-scale magnet arrays of the Prototype Small-Gap Undulator (PSGU) under contract for the NSLS, while he was employed at Rocketdyne, now part of Boeing, in California. After returning to BNL in 1993, Rakowsky worked with Lynch, Stefan and other collaborators at SPring8 in Japan, to develop the breakthrough In-Vacuum Undulator (IVUN).

Lynch and Rakowsky then developed the third-generation, higher-performance Mini-Gap Undulator (MGU), which has operated in the x-ray ring since January 2002. The MGU's compact size will allow the installation of two more MGUs, resulting in two new undulator beamlines. The next MGU was installed in May to serve a new beamline funded by the National Institutes of Health and dedicated to structural biology.

In developing these devices, still record-breakers as the smallest in their field, Rakowsky led the way in magnetic design and measurement, while Lynch provided the mechanical solutions to meet each challenge.

Their success has established the NSLS as the leader in small-gap undulator technology.

This innovation has dramatically changed light-source development world-wide to favor medium-energy machines of around 3-GeV over the more costly 6-to-8-GeV machines previously constructed as hard x-ray sources. The small-gap concept also serves as the basis for the NSLS 3-GeV upgrade proposal.

—Patrice Pages

BNL/Canadian Space Agency Experiment Lost in Tragic Columbia Accident

February 1, 2003

Among the 80 scientific experiments lost in the recent tragic accident involving the space shuttle Columbia, one was prepared by a team of scientists who would have studied the results at the NSLS. This experiment had been designed to provide insights into developing not only new drugs against cancer, but also drought-resistant plants.

The experiment is part of the Canadian Space Agency's Protein Crystal Growth (PCG) program, a project aimed at studying how proteins grow in space. A series of different protein crystal growth experiments were conducted in the recent Columbia mission, one of which is involved in a hallmark of cancer, called cachexia, which results in emaciation and muscle wasting. Another represented a protein used by plants to defend against environmental stress, such as drought and high salinity.

Protein crystal growth experiments were conducted on the Space Shuttle Columbia.

"Previous crystal growth experiments conducted in space have provided evidence that, when protein crystals grow in space, they are of higher quality, that is, they have fewer defects and may be of larger size than those grown on Earth," says Robert Sweet, BNL Biology Department and a PCG project collaborator. "But we know too little about the effectiveness of having crystals grow in space, where there is nearly no gravity. So we decided to compare crystals grown on Earth and in space."

Controlled Experiment

The PCG scientists, led by Jurgen Sygusch, a biochemist at the University of Montreal in Canada, designed a controlled experiment wherein duplicate samples of proteins in solution were taken to Kennedy Space Center (KSC), and one set of samples was taken into space on the space shuttle Columbia, one of five reusable spacecraft that had been employed by the National Aeronautics & Space Administration (NASA) since 1981.

While Columbia circled Earth from January 16 to 31, the protein samples on board were allowed to crystallize. At the same time, duplicate samples acting as controls were crystallized at KSC in the same type of crystallization hardware. If Columbia had returned to Earth, the two sets of samples were to have been compared with one another, using the intense light generated by the NSLS.

How Crystals Grow

"When crystals grow on Earth, gravity introduces disturbances that affect the way protein molecules assemble into a crystal lattice," Sygusch says. "But, in spacecraft such as Columbia, which orbit about 400 miles above Earth, crystal growth is subjected to only a millionth of Earth's gravity — called microgravity — so that the protein molecules during crystal growth are free of these disturbances, which is what we wanted to study."

In their experiment, the scientists planned to investigate two important disturbances, called convection and sedimentation, that occur during crystal growth.

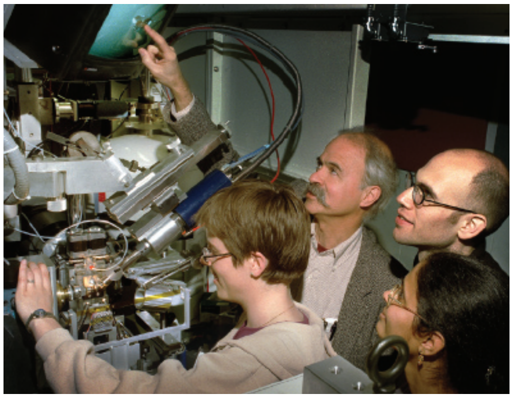
"You can imagine a growing crystal being like layers of molecules that are piling up on each other," Sygusch says. "Non-crystalline aggregations of protein molecules that form naturally under crystal-growth conditions tend to disturb the deposition of these layers."

Sygusch explains that convection in the protein solution occurs because the protein is actually at a lower concentration, and therefore lower density, immediately adjacent to the growing crystal. This causes an upwelling of the solution that causes a surprisingly vigorous mixing of proteins and their aggregates.

As for sedimentation, once small crystallites have formed, they tend to sink to the bottom of the container, out of the zone of where they grew, and often cease growth.

Both convection and sedimentation affect crystal growth on Earth. In micro-gravity, the absence of convection eliminates solution mixing and reduces incorporation of protein aggregates into the crystal lattice, while no sedimentation allows crystallites to grow into larger crystals, leading to crystals of better quality. The higher crystallinity translates into a better definition of the atomic detail that can be obtained from synchrotron diffraction experiments.

"To get our results," Sweet says, "we had not only prepared two identical experiments, one on Earth and the other in space, but we had also decided that the data collected from both experiments would be analyzed in a double-blind fashion. After crystals had been recovered from Columbia, they would have been combined with the crystals grown on Earth in such a way that we could measure results without knowing until afterwards from which source the crystals came."



BNL scientist Bob Sweet demonstrates protein crystallography experiments underway at the NSLS.

Next Step

The PCG team is now waiting to know when they will be able to send new experiments in space, perhaps in an unmanned spacecraft. "Our experiment could easily be activated automatically," comments Sweet.

"Some perturbations, such as astronaut movements and orbit corrections, could be avoided if this experiment were performed in a free flyer or unmanned spacecraft," agrees Sygusch, who is confident that more chances to conduct experiments in space will arise in the near future.

"The latest Columbia mission was a tragic human disaster and a scientific catastrophe," Sygusch says. "But, once we understand what happened, we must continue. We will be able to investigate something never achieved before in the history of humankind: a glimpse of what life might look like in a world with a different level of gravity."

—Patrice Pages